Chemical Engineering and Processing 105 (2016) 73–78

Contents lists available at ScienceDirect

Chemical Engineering and Processing: Process Intensification

journal homepage: www.elsevier.com/locate/cep

Magnetite and anthracite assisted microwave heating flue gas desulfurization gypsum

Zhong-hai Ji, Jin Chen*, Yu Guo, Li-na Guo, Wei Li

College of Materials Science and Engineering, Taiyuan University of Technology, Taiyuan, Shanxi 030024, China

ARTICLE INFO

Article history:
Received 17 November 2015
Received in revised form 25 January 2016
Accepted 30 January 2016
Available online 25 April 2016

Keywords:
Microwave heating
FGD gypsum
Anthracite
Magnetite
Heating rate

ABSTRACT

The heating characteristics of flue gas desulfurization (FGD) gypsum and their mixtures have been investigated with a microwave reactor. The influences of three factors(water in FGD gypsum and the additives, anthracite and magnetite), on the microwave heating are the focuses of this work. The heating curves revealed that the temperature of FGD gypsum only reached to 299 °C after microwave heating for 90 min. With increasing proportions of magnetite and/or anthracite, the final temperature of sample rose significantly. The peaks of the heating rates between magnetite and anthracite are partially overlapped. Adding both of them to FGD gypsum, continuous increase of temperature was achieved. When adding 10 wt% magnetite and 8 wt% anthracite to FGD gypsum, a relatively high heating rate was obtained. The heating rate is intensified, in sequence, by water, magnetite, burning of anthracite and hot spots caused by agglomerates of anthracite and magnetite. Within 82 min of microwave radiation, temperature of the mixture rose to 1040 °C which met the decomposition temperature of FGD gypsum.

1. Introduction

Decomposition of FGD gypsum, $CaSO_4{\cdot}2H_2O$ as its main component, is one of the most promising recycle routes for recovering SO_2 and reusing the residual solid waste [1–3]. To obtain more valuable targeted-products and increase concentration of SO_2, recent interests are focusing on adding catalysts like Fe_2O_3, $FeCl_3$ and/or finding optimum weight radio of gypsum and high-sulphur concentration coal and so forth [4–9].

Microwave heating, highlighted by virtue of volumetric heating and selective heating in contrast with the conventional heating [10], is a promising technology which has growing interest and considerable potential for various applications in mineral processing [11], environmental engineering [12], leaching [13], extractive metallurgy [14] and so forth. However, previous authors emphasized more on heating up high dielectric property materials directly while few papers had mentioned microwave heating low loss materials or microwave transparent materials.

Report [15] showed that calcium sulfate dehydrate ($CaSO_4$) is almost transparent to microwaves. Thus, so far, fewer reports of systematic study have characterized microwave heating of mixtures involving FGD gypsum. However, this problem has been worked out in our recent heating method by adding magnetite and anthracite to assist microwave heating. Anthracite can remarkably lower the decomposition temperature of $CaSO_4$ by burning off to provide a reducing ambient and reacting with $CaSO_4$ [16]. In the decomposition process, temperature is one of the most important factors which had been repeatedly certified in the previous published papers [4–9]. Aimed to find an economic and feasible way, in this study, different weight radios of anthracite and magnetite were added to FGD gypsum. The change trends of the samples' microwave heating characteristics were investigated. And an optimal combination of FGD gypsum mixed with anthracite and magnetite for a high temperature as well as a high conversion rate was explored.

2. Experimental materials and methods

2.1. Sample preparation

FGD gypsum was prepared from Taiyuan Iron and Steel Corporation in China. Its chemical compositions are listed in Table 1. For comparison, anhydrous gypsum had been prepared by a muffle furnace for 5 h at 300 °C. Table 2 shows the chemical analysis of anthracite (Yangquan, China). Xuanhua magnetite ore fine is presented in Table 3. The samples used were crushed and grinded to coarse powder with particle sizes of less than 147 μm. The relative complex permittivity and permeability were

* Corresponding author.
E-mail address: chenjin2013815@126.com (J. Chen).

http://dx.doi.org/10.1016/j.cep.2016.01.019
0255-2701/

Table 1
chemical composition of Taigang FGD gypsum (mass%).

Material	CaO	SO_3	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	Na_2O	H_2O^+
FGD gypsum	31.47	43.05	1.72	1.32	0.53	0.16	0.12	0.037	20.03

Table 2
chemical composition of Yangquan anthracite (mass%).

Material	A_{ad}	V_{ad}	M_{ad}	S_{ad}	FC_{ad}
Anthracite	14.26	7.19	0.92	0.69	76.94

A_{ad}, ash in the air dried sample; V_{ad}, volatile matter in the air dried sample; M_{ad}, moisture in the air dried sample; S_{ad}, sulfur in the air dried sample, and FC_{ad}, fixed carbon in the air dried sample.

An adjustable microwave oven with multimode resonant cavities was designed which contained 12 magnetrons, installed symmetrically, and each with 0.8 kW of power at a fixed frequency of 2.45 GHz, as shown in Fig. 1. The crucible was placed in the center of the microwave cavities where the mixtures could be radiated uniformly. Since the volume ratio of the crucible to the cavities is only 1/1000, the quality factor of the microwave cavities remains little change. A K-type thermocouple (0–1300 °C), shielded with a stainless pipe, was inserted into the center of the sample to continuously measure the sample's interior temperature. The measurements were recorded by a digital-display temperature conditioner (XMT-101). Without protective gas, the mixed materials were heated up by microwave oven under atmospheric pressure.

Table 3
chemical composition of Xuanhua magnetite ore fines (mass%).

Material	Fe_3O_4	FeS	CaO	MgO	SiO_2	Al_2O_3	K_2O	Na_2O	MnO_2	TiO_2	S
Magnetite	88.51	0.079	0.24	0.49	5.91	0.38	0.062	0.056	0.16	0.76	0.45

Table 4
Electromagnetic parameters of magnetite ore fines and anthracite at 2.45 GHz of microwave frequency at 25 °C.

Sample	ε'	ε''	$\tan\delta_E$	μ'	μ''	$\tan\delta_M$	$\sigma/(\Omega^{-1}\,cm^{-1})$
Magnetite	9.92	0.11	0.0111	1.55	0.31	0.20005	1.54×10^{-4}
Anthracite	36.85	10.65	0.029	0.97	−0.03	−0.031	1.448

measured by a vector network analyzer (HP-8722ES, USA) at 25 °C in the frequency of 2.45 GHz and were presented in Table 4. Table 3 and Table 4 have been reported in the previous paper in our group [17].

2.2. Experimental setup and procedure

In order to obtain a homogeneous sample, FGD gypsum, anthracite and magnetite powders were thoroughly mixed of about 1 kg by a grinding miller in different weight ratios and naturally placed into a wave-transparent fire-clay crucible. The crucible, containing 65 mm in diameter and 260 mm in height, is made up of light firebricks which do not interact with microwaves.

2.3. Characterizations

The components of the partially reacted residues were analyzed with the Cu Kα line on a Rigaku D/max 2500 X-ray diffractometer (XRD) with patterns recorded in a range of 20–80°. The desulfurization rates of the samples were measured by a microcomputer rapid sulfur tester (WDL-100C) and calculated indirectly by the test date of SO_2 released from the reacted residues.

2.4. Microwave heating fundamentals

The absorption of microwaves is related to permittivity (ε) and permeability (μ) which can be defined as follows:

$$\varepsilon^*{}_r = \varepsilon'_r - i\varepsilon''_r \quad \mu^*{}_r = \mu'_r - i\mu''_r \tag{1}$$

$$\tan\delta_\varepsilon = \frac{\varepsilon'_r}{\varepsilon'_r} \quad \tan\delta_\mu = \frac{\mu'_r}{\mu'_r} \tag{2}$$

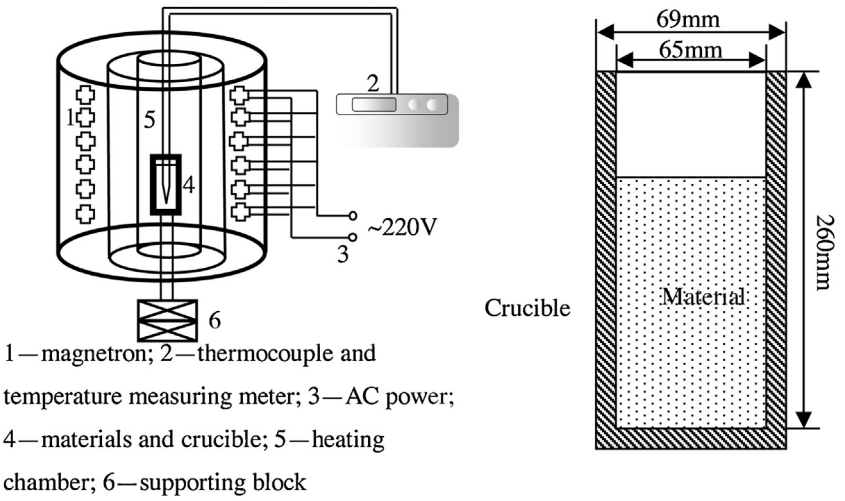

Fig. 1. Schematic diagram of test apparatus for microwave heating.